Solid layer thermal-conductivity measurement techniques

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The thermal conductivities of solid layers of thicknesses from 0.01 to 100 µm affect the performance and reliability of electronic circuits, laser systems, and microfabricated sensors. This work reviews techniques that measure the effective thermal conductivity along and normal to these layers. Recent measurements using microfabricated experimental structures show the importance of measuring the conductivities of layers that closely resemble those in the application. Several promising non-contact techniques use laser light for heating and infrared detectors for temperature measurements. For transparent layers these methods require optical coatings whose impact on the measurements has not been determined. There is a need for uncertainty analysis in many cases, particularly for those techniques which apply to very thin layers or to layers with very high conductivities.

CONTENTS

NOMENCLATURE

C = specific heat per unit volume, $J\ m^{-3}\ K^{-1}$

d = layer thickness, m

d_{sub} = substrate thickness, m

d_T = thickness of thermocouple bridge, m

E = energy deposited by laser, J

E'' = energy deposited per unit area, $J\ m^{-2}$

h = heat transfer coefficient, $W\ m^{-2}\ K^{-1}$

i = integer in summation, Eq (11)

K_0 = modified Bessel function of first kind of order zero

k = thermal conductivity, $W\ m^{-1}\ K^{-1}$

$k_{a,eff}$ = effective thermal conductivity for conduction along layer, $W\ m^{-1}\ K^{-1}$

k_{app} = apparent thermal conductivity measured by thermal comparator, $W\ m^{-1}\ K^{-1}$

$k_{n,eff}$ = effective thermal conductivity for conduction normal to layer, $W\ m^{-1}\ K^{-1}$

k_{sub} = substrate thermal conductivity, $W\ m^{-1}\ K^{-1}$

k_T = thermocouple bridge thermal conductivity, $W\ m^{-1}\ K^{-1}$

L = length, m

L_0 = Lorenz number = $2.45 \times 10^{-8}\ W\ \Omega\ K^{-2}$

l = half-width of laser line, m

Q = heat flow, W

q = heat flux, $W\ m^{-2}$

q_a = amplitude of periodic heat flux, $W\ m^{-2}$

q_{al} = heat-flow amplitude per unit length, $W\ m^{-1n}$

q_x = heat flux in x direction, along layer, $W\ m^{-2}$

q_y = heat flux in y direction, normal to layer, $W\ m^{-2}$

R_{app} = apparent thermal resistance, $m^2\ K\ W^{-1}$

R_{sub} = substrate thermal resistance, $m^2\ K\ W^{-1}$

R_T = thermal resistance, $m^2\ K\ W^{-1}$

r = distance from heating source, m

r_c = heat-flow radius, m

T = temperature, K

ΔT = temperature difference, K

T_a = amplitude of periodic temperature, K

T_A = temperature of bridge A, K

T_B = average substrate temperature below bridge A, K

T_i = initial layer temperature due to pulse heating, K

T_0 = boundary or reference temperature, K

T_1 = boundary temperature, K

t = time, s

t_L = duration of laser pulse, s

U_T = ratio of temperature changes

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w = width, m

x = coordinate in plane of substrate, m

x_s = separation between bridges, m

y = coordinate normal to substrate, m

Greek Symbols

α = thermal diffusivity, $m^2 \, s^{-1}$

ε = emissivity

λ_T = inverse thermal diffusion length = $(\omega/\alpha)^{1/2}$, m^{-1}

ρ_e = electrical resistivity, $\Omega \, m$

ω = angular frequency, rad s^{-1}

1. INTRODUCTION

The thermal conductivities of thin solid layers are needed for the design of field-effect transistors in electronic circuits, coated lenses in laser systems, and microfabricated superconducting radiation detectors (Goodson and Flik, 1992; Guenther and McIver, 1988; Verghese et al, 1992). The thermal conductivity of a layer can differ from that of a bulk sample of the same material for two reasons. Layer fabrication techniques, such as chemical-vapor deposition (CVD), usually result in a microstructure or purity in the layer that differs from that in the bulk material, which influences the thermal conductivity. In addition, the small thickness of the layer can increase the importance of interfacial effects such as thermal boundary resistances and the boundary scattering of heat carriers, electrons and phonons. This can reduce the effective conductivity of the layer. Transmission electron microscopy, micro-Raman spectroscopy, and x-ray and electron diffraction provide insight into the microstructure of layers. But the precise microstructural information, e.g., the size and orientation of grains and the density of point defects, needed for predictions of the thermal conductivity is often not available. This makes accurate techniques for measuring the conductivity of layers essential.

Cahill et al (1989) reviewed several techniques which measure the thermal conductivity in the direction normal to layers. These authors made helpful observations about the effects of phonon-boundary scattering and interfacial layers on the measurements at cryogenic temperatures. Some of these insights are presented in greater detail in the review of research on thermal boundary resistance by Swartz and Pohl (1989). Graebner (1993) described techniques that measured the thermal conductivity of CVD diamond layers and compared the resulting data. There remains a need for a review which includes techniques used for other layers, especially the thin, low-conductivity amorphous layers that coat lenses and serve as passivation in circuits. Guenther and McIver (1988) and Lambropoulos et al (1991) surveyed the existing data for the thermal conductivity in the direction normal to amorphous dielectric layers, but in most cases did not provide the details of the measurement techniques used to obtain these data. As a result, it is often difficult to assess the accuracy of the techniques or their applicability to layers

which have different thicknesses or are made of different materials.

This work helps to remedy this situation by reviewing layer thermal-conductivity measurement techniques. The layer geometry is depicted in Fig 1, which defines the coordinates x and y to be along and normal to the layer, respectively. The thermal conductivity measured in layers is not necessarily a property of the layer material. Due to heat-carrier boundary scattering or thermal boundary resistances, the apparent thermal conductivities of layers often depend on the direction of heat flow, even for isotropic materials, the layer thickness, and the properties of the layer boundaries. For this reason, most measurements yield an *effective* thermal conductivity, which is valid only for a given layer thickness and direction of heat flow. The effective thermal conductivity along a layer is

$$k_{a,eff} = -q_x \left[\frac{dT}{dx}\right]^{-1} \qquad (1)$$

where T is the local layer temperature, which is assumed not to vary in the y direction, and q_x is the heat flux in the x direction in the layer, averaged in the y direction. The thermal conductivity normal to a layer is defined so that it accounts for the volume resistance of the layer *and the thermal resistances between the layer and the bounding media,*

$$k_{n,eff} = q_y \frac{d}{T_0 - T_1} \qquad (2)$$

where the temperatures T_0 and T_1 are those of the bounding media just outside of the interfaces with the layer, d is the layer thickness, and q_y is the heat flux in the y direction. For highly-conductive layers, the in-plane conductivity $k_{a,eff}$ is important because these layers govern lateral heat conduction in multilayer structures, e.g., the silicon and aluminum thin-layer bridges in silicon-on-insulator circuits (Goodson and Flik, 1992; Goodson et al, 1993c). For layers made of materials which are poor thermal conductors,
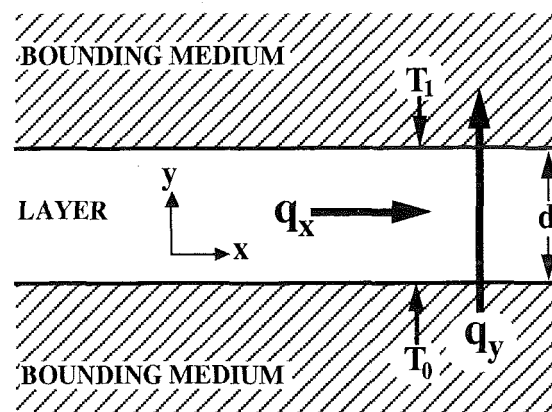

FIG 1. Schematic of the layer geometry.

e.g., amorphous materials, $k_{n,eff}$ is of greatest importance because these layers dominate the thermal resistance in the y direction. Recently, amorphous thin-layer membranes were used to thermally isolate radiation detecting elements (Verghese et al, 1992), providing an important exception to this rule. For these membranes, the in-plane conductivity $k_{a,eff}$ governs the thermal conductance and therefore the sensitivity and time constant of the detector.

Layer thermal conductivity measurement techniques are distinguished by the time-dependence and the source of the heating they employ, as well as their method of temperature measurement. Steady-state techniques induce a time-independent heat flux, measure a resulting temperature difference or distribution, and calculate the thermal conductivity. Transient techniques induce a time-dependent heat-flux, e.g., an impulse or periodic function, and in most cases determine the thermal diffusivity α by comparing analytical solutions to the transient thermal-conduction equation with the measured time-dependent temperature. The thermal conductivity can be obtained using $k = \alpha\ C$, where C is the bulk specific heat per unit volume. Nonporous layers of thickness and grain size large compared to the wavelength of energy carriers possess the same energy-carrier densities of states and therefore the same specific heat per unit volume as bulk materials. Heating mechanisms include Joule heating, e.g., due to electrical conduction in a bridge deposited on the sample, and the absorption of laser radiation. Temperature-measurement tools include thermocouples, infrared (IR) pyrometers, and electrical-resistance thermometers.

This review separates the techniques into two basic groups. Section 2 discusses techniques which measure $k_{a,eff}$, and Section 3 discusses techniques which measure $k_{n,eff}$. The techniques are further divided into groups according to the time dependence of the heating source which is used. Sections 2.1 and 3.1 review steady-state techniques, and Sections 2.2 and 3.2 review transient techniques.

2. CONDUCTIVITY ALONG LAYERS

2.1 Steady-State Techniques

Several of the techniques discussed here were applied both to free-standing layers and to layers on substrates with low thermal conductivities, e.g., lead layers on amorphous dielectric substrates. This is appropriate when the ratio $d\ k_{a,eff}/ (d_{sub}\ k_{sub})$ is of the order of or larger than unity, where d_{sub} is the substrate thickness and k_{sub} is the substrate thermal conductivity. The temperatures in the layer and substrate are assumed not vary in the plane normal to the direction of heat flow. The total rate of heat flow is the sum of the heat flow rates due to the layer and the substrate. For the case of steady-state conduction of the power Q in the direction x along a layer-substrate composite bridge of width w, the layer conductivity is

$$k_{a,eff} = -\frac{Q}{w\,d}\left[\frac{dT}{dx}\right]^{-1} - \frac{d_{sub}}{d}k_{sub} \qquad (3)$$

In what follows, a "bridge" is a layer or a layer-substrate composite with a finite cross-sectional area that is long in the direction normal to the cross section. A "free-standing bridge" consists of a layer without a substrate or a layer-substrate composite for which Eq (3) was used.

The most common technique was applied to free-standing bridges at temperatures from 5 to 450 K. The bridge was attached to an isothermal heat sink at the temperature T_0 in a vacuum chamber, as shown in Fig 2. Attached to the opposite end of the bridge, which was mechanically unsupported, was an electrical-resistance heater. The two junctions of a thermocouple were attached to the bridge, one near the free end and the other near the heat sink. The thermocouple measured the temperature difference ΔT which occurred over the length L along the bridge. The thermal conductivity $k_{a,eff}$ was determined using Eq (1) with $q_x = Q/(wd)$ and $-dT/dx = \Delta T / L$, where Q is the heating power and w is the bridge width. This technique was used for lead bridges by Pompe and Schmidt (1975) between 5 and 20 K. More recently, Morelli et al (1988) and Graebner et al (1992a) used this technique to measure the thermal conductivity of diamond bridges between 10 K and room temperature. The primary causes of experimental error in this technique are conduction through the thermocouple and heater wires, which can be minimized by using very thin wires, and radiation from the heater. Pompe and Schmidt (1975) and Graebner et al (1992a) used a heater near the heat sink, shown in Fig 2, to investigate the magnitude of this error. When the heater near the heat sink generates heat and the heater near the tip is off, the temperature drop along the bridge can be used to estimate the energy flow out of the bridge due to radiation and conduction from the wires. Graebner et al (1992a) used several thermocouples along the length of the sample to more accurately estimate the heat flow. This allowed them to achieve an uncertainty of a few percent, which was among the best below room temperature.
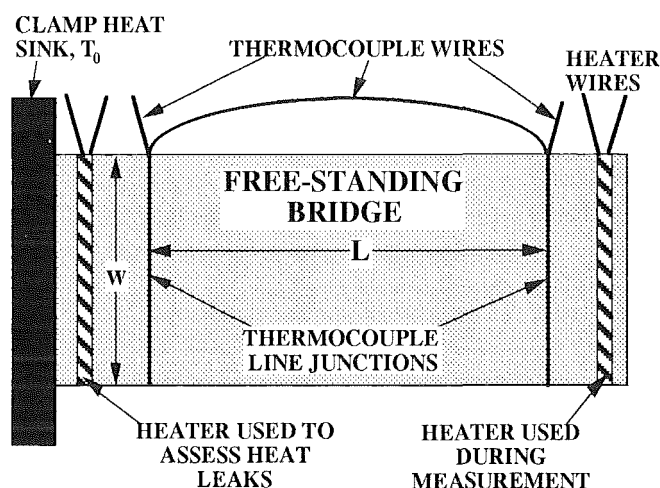
FIG 2. Experimental structure used by Pompe and Schmidt (1975) to measure the thermal conductivity along a bridge.

The technique of Nath and Chopra (1973) is similar. These authors attached the cold end of the bridge to an isolated block with a known heat capacity. The other end of the bridge was secured to an isothermal heater. A thermocouple measured the temperature difference between the heater and the block. The rate of heat flow through the bridge Q was calculated from the measured block temperature and the conductance of the block to the surrounding structure due to radiation. The conductance was calculated from the known heat capacity of the block and the exponential decay with time of the block temperature when it was allowed to cool. The accuracy of this technique is reduced by radiation from the bridge and conduction through the cold thermocouple junction. In contrast to the technique used by Pompe and Schmidt (1975), the technique of Nath and Chopra (1973) is not influenced by radiation from the heater or conduction due to the warm thermocouple junction. Nath and Chopra (1973) did not give the experimental uncertainty. They used this technique for copper layers on amorphous dielectric substrates above room temperature. This technique demands a conductance from the block due to radiation that varies little for the temperature differences used between the block and the surrounding structure. This condition is most easily satisfied above room temperature.

Nath and Chopra (1973) developed a complementary low-temperature technique which uses a bare-substrate bridge and a layer-substrate composite bridge, each attached to blocks of identical heat capacity. The differences between the temperatures of the blocks and the cooling bath temperature were small and radiation was neglected. Both substrates were assumed to possess the same thermal conductivity and cross-sectional area. At time $t = 0$, the free ends of both bridges were attached to a warmer block, which was assumed to remain isothermal while cooling through the two samples. The heat capacity of the free-standing bridge was neglected. One-dimensional conduction analysis determined $k_{a,eff}$ in the layer from the substrate dimensions, the identical heat capacities of the cooler blocks, and the transient temperatures

FIG 3. Self-heated bridge structure used by Boiko et al (1973).

of the three blocks. This technique yields the conductivity independently from the substrate conductivity, so that Eq (3) is not required. Although this technique involves a transient response, it is discussed in this section because the heat capacity in the layer is neglected.

Boiko et al (1973) and Völklein and Kessler (1984) developed similar techniques which measured both the thermal conductivity and the radiation emissivity of free-standing electrically-conducting bridges. The technique of Boiko et al (1973) was applied to metal bridges from 300-900 K, and the technique of Völklein and Kessler (1984) was applied to semimetal bridges from 80 to 400 K. In both cases, the bridge was suspended between two heat sinks at the temperature T_0, as shown in Fig 3. Both techniques employed Joule heating in the bridge to induce heat flow, and solved the one-dimensional thermal-conduction equation in the bridge considering Joule heating and radiation from the bridge.

Boiko et al (1973) measured the bridge temperature at seven locations along its length using the temperature dependence of the lattice parameter of the bridge material, which was measured using electron diffraction. The change in the lattice parameter was assumed to be proportional to the product of the temperature change and the coefficient of thermal expansion. The reported uncertainty in temperature changes measured this way was +/- 5 K. As a result, large temperature changes were employed, varying between 50 and 720 K for one set of measurements. The authors determined the values of the temperature-independent thermal conductivity $k_{a,eff}$ and the emissivity which were consistent with the solution to the energy equation and the measured temperature distribution. The large temperature changes required by this technique render it inappropriate for materials whose thermal conductivities vary substantially with temperature, e.g., semiconductors and semimetals above room temperature.

Völklein and Kessler (1984) used much smaller temperature changes and calculated the average thermal conductance from the bridge, i.e., the ratio of the Joule heating power dissipated in the bridge to its average temperature change, from current-voltage data. The change in the average bridge temperature from the reference temperature T_0 is approximately proportional to the change of the bridge electrical resistance from its resistance when isothermal at T_0. Using the energy equation, the authors predicted the average thermal conductance from the bridge as a function of its thermal conductivity and emissivity, which were both assumed to be independent of temperature. By measuring the thermal conductance from two bridges with different lengths and identical thermal properties, the thermal conductivity and emissivity were obtained independently. The error due to the use of a temperature-independent emissivity was not determined. This approximation needs to be examined for metals, whose normal total emissivity is nearly linearly proportional to temperature above room temperature (Siegel and Howell, 1981). This technique requires two layers of identical thermal properties. But if the emissivity of a layer is known, the thermal conductivity can
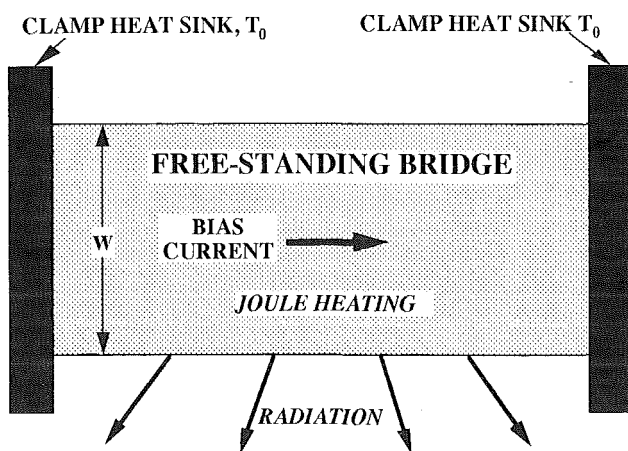
be calculated from measurements performed only on that layer. This technique should only be used when radiation losses are large compared to the energy conducted along the layer, which can occur for very-thin free-standing bridges.

Recently, Graebner et al (1992c), Tai et al (1988), and Völklein and Baltes (1992) used novel, microfabricated experimental structures to measure the thermal conductivity of thin layers. Graebner et al (1992c) etched the substrate from underneath rectangular 2 x 4 mm^2 sections of diamond layers of thickness between 2.8 and 13.1 μm. They deposited a heater bridge and thermocouple bridges on the top of each diamond rectangular layer section, as shown in Fig 4. The boundaries of the suspended layer were assumed to be isothermal at the heat-sink temperature, T_0. The validity of this assumption for the case of the thickest highly-conductive diamond layers must be investigated. Comparison of the measured temperature profile with that predicted by a numerical solution of the two-dimensional thermal conduction equation yielded $k_{a,eff}$ in the diamond layers.

Tai et al (1988) suspended heavily-doped, 1.5 μm thick polysilicon (polycrystalline silicon) bridges of lengths from 100 to 200 μm between phosphosilicate glass supports. These structures resembled microfabricated flow sensors whose thermal design requires the conductivity of the doped silicon bridges. Each bridge had a short, lightly-doped center segment whose electrical resistance dominated that of the entire bridge. A bias current induced heat flow from the lightly-doped segment, and a solution to the one-dimensional heat-conduction equation yielded the conductivity of the bridge from its measured electrical resistance, which depended on temperature. Völklein and Baltes (1992) suspended a heavily-doped polysilicon sheet of thickness 0.37 μm on silicon-dioxide passivation above a silicon substrate. Metal heater and thermometer bridges were deposited on the sheet far from its contact with the substrate. Most of the heat

generated in the heater bridge was conducted to the substrate through one-dimensional conduction in the polysilicon. The other contributions to the heater-to-substrate conductance were subtracted using data from a simultaneous measurement on a nearly-identical microfabricated structure without the polysilicon. This allowed the polysilicon conductivity to be calculated. The measurement techniques of Tai et al (1988) and Völklein and Baltes (1992) yielded data appropriate for the design and analysis of microfabricated sensors.

Dua and Agarwala (1972) used the Wiedemann-Franz law (Kittel, 1986),

$$k_{a,eff} = \frac{L_0\,T}{\rho_e} \qquad (4)$$

where L_0 is the Lorenz number, to estimate the thermal conductivity of metal layers from the measured electrical resistivity ρ_e. Equation (4) is valid at temperatures above the Debye temperature, which is near 100 K for most metals, or at temperatures where defect- or boundary-scattering dominates. The second condition is satisfied below about a fifth of the Debye temperature for nearly-pure metals and at higher temperatures for impure metals and very thin layers. Equation (4) is accurate within 14 percent for common metals at room temperature (Kittel, 1986).

2.2　Transient Techniques

Mastrangelo and Muller (1988) used experimental structures similar to those of Tai et al (1988), i.e., a heavily-doped polysilicon bridge suspended between glass supports. In this case the bridges were uniformly doped and were 1.3 μm thick and of length between 180 and 280 μm. A bias current in the layer induced Joule self-heating. The authors solved the transient one-dimensional thermal-conduction equation in the bridge, which accounted for its temperature-dependent electrical resistivity and neglected radiation. The thermal diffusivity was obtained by comparing the predicted time-dependent electrical-resistance response with the measured response. Using the bulk specific heat, the measured thermal diffusivity values agreed well with the thermal conductivity measured in similar structures by Tai et al (1988).

Hatta (1985) developed the technique illustrated in Fig 5. A portion of a free-standing rectangular layer was masked from a sheet of laser light with periodic heat flux in the y direction, $q_y = q_a\,(1 + \sin\omega t\,)$, where q_a is the amplitude of the absorbed heat flux and ω is its angular frequency. On the layer under the mask was a thermocouple junction separated from the mask edge by the distance x. A solution to the transient one-dimensional thermal-conduction equation neglecting heat transfer from the layer yields the approximate amplitude T_a of the periodic component of the temperature at location x,

FREE-STANDING
FILM BOUNDARY AT T_0

THERMOCOUPLE
BRIDGES

HEATER BRIDGE

THERMOCOUPLE
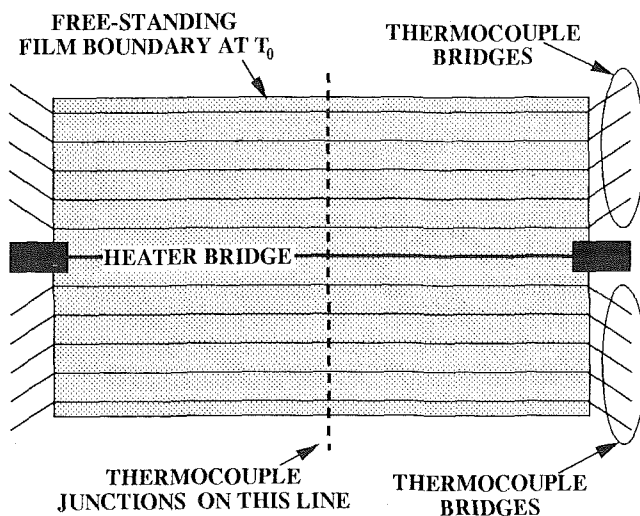JUNCTIONS ON THIS LINE

THERMOCOUPLE
BRIDGES

FIG 4.　Novel experimental structure of Graebner et al (1992c) for measuring $k_{a,eff}$ of diamond layers. The substrate is etched from beneath the rectangular layer section shown.

$$T_a\,(x) = \frac{q_a}{2\,\omega\,C\,d}\,\exp\!\left(-\frac{\lambda_T\,x}{\sqrt{2}}\right) \qquad (5)$$

The inverse thermal-diffusion length is $\lambda_T = (\omega/\alpha)^{1/2}$, where α is the thermal diffusivity in the x direction. The distance between the thermocouple and the mask edge can be changed by moving the mask. Equation (5) and the measured function $T_a(x)$ yield α. The frequency must be small enough so that the thermal diffusion length is much larger than the layer thickness. Subsequent research investigated the influence of the thermocouple on the temperature in the layer (Hatta et al, 1986) and optimized the sample dimensions for the measurement (Hatta et al, 1987). The authors did not investigate the potential of a microfabricated thermocouple, such as those employed by Graebner et al (1992c), for measuring temperature without disturbing conduction in the layer.

Similar techniques developed by Visser et al (1992) and Shibata et al (1991) avoided the influence of the thermocouple on conduction in the layer by using infrared thermography to measure the layer temperature. Visser et al (1992) used a highly-focussed, periodic laser beam to heat a spot with diameter near 10 µm on a layer of large dimensions. A solution to the transient one-dimensional thermal-conduction equation with cylindrical coordinates yields the phase and amplitude of the periodic temperature in the layer as functions of its diffusivity α, heat capacity per unit volume C, the distance from the focus, and the heat transfer coefficient h from the layer surface to the ambient temperature. Fitting the measured amplitude and phase of the temperature fluctuations to the predictions yields α and h. In the techniques of Hatta (1985) and Visser et al (1992), the phase of the temperature fluctuations yields the thermal diffusivity without requiring the heat transfer coefficient h. The primary advantage of the technique of Visser et al (1992) is that it does not require physical contact with the sample layer. The method of Hatta (1985) could also be applied using infrared thermography.

Shibata et al (1991) employed a brief line pulse of laser light of time duration t_L and spatial width $2l$ to irradiate a free-standing rectangular layer section. The line was incident on the entire width w of the layer section at its center, inducing one-dimensional heat conduction along its length. The half-width l and the time duration t_L of the laser-pulse line satisfied $l^2 >> \alpha\, t_L$. This allowed the transient temperature in the layer to be calculated by assuming an initial temperature change $T_i - T_0 = E / (2l\, d\, w\, C)$ in the irradiated portion of the layer, where E is the total energy deposited. Heat transfer from the layer was neglected. The temperature was detected from the bottom of the layer at a distance from the heat source which was large compared to the layer thickness. Comparison of the time required for the measured temperature to reach half of its maximum value with the analytical prediction of this time yielded the thermal diffusivity. The impact of neglecting heat-transfer from the layer needs to be assessed.

Photothermal displacement spectroscopy at transient thermal gratings is based on the principle of Eichler et al (1971; 1973), who used the interference of two pulsed laser beams at the same frequency with different angles of incidence on a sample to deposit energy with a density that varied periodically in one lateral dimension. The technique of Harata et al (1990) and Käding (1993a) generates heat at the surface of the sample layer. The thermal expansion of the layer due to the brief laser pulse results in a spatially-periodic displacement of the surface, whose exponential decay with time is governed by the lateral thermal diffusivity of the layer within a depth comparable to the spatial period of the grating. The time dependence of the deflection of a probe laser beam yields the relaxation time of the displacement, from which the diffusivity is calculated. The depth of observation in the sample can be controlled by varying the spatial period of the grating. Käding et al (1993a; 1993b) provided theory for this method that facilitated diffusivity measurements in CVD diamond layers within a depth as small as 10 µm. This method has the advantage of being local in three dimensions with a lengthscale comparable to the grating period. The uncertainty of data obtained using this technique must be determined, particularly for measurements on layers of very high thermal diffusivity.

The optical techniques require the deposition of a thin absorbing coating on transparent or semi-transparent layers. If these coatings are too thick, they influence the diffusivity measurement. For coatings of low thermal diffusivity, the heat cannot diffuse through the coating during the relevant time interval, e.g., one period of the laser flux. This problem can be aggravated by a thermal resistance between the coating and the layer. If the diffusivity of the coating is too high, it may contribute to transport in the plane of the layer. Visser et al (1992) compared values of the thermal diffusivity of measured in copper sheets of thickness 100 µm that were coated with different materials. Their data show that the coating material can strongly influence the measured diffusivity. The influence of the coating material on the diffusivity measurement increases with decreasing layer thickness. Further investigation needs to determine the appropriateness of optical techniques for measuring the

PERIODIC RADIATION FLUX,
ANGULAR FREQUENCY ω

MOVABLE MASK

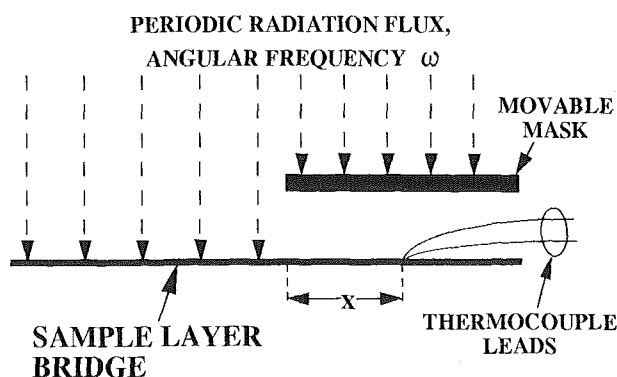SAMPLE LAYER BRIDGE

THERMOCOUPLE LEADS

X

FIG 5. Side view of the experimental structure of Hatta (1985) for measuring the thermal diffusivity along layers heated by a sheet of laser light. The mask was moved to change the distance x while the thermocouple junction remained fixed.

thermal diffusivity of very-thin layers with low absorbtances.

3. CONDUCTIVITY NORMAL TO LAYERS

3.1 Steady-State Techniques

The techniques described in this sub-section apply only to layers on substrates satisfying $k_{n,eff} << k_{sub}$. Goldsmid et al (1983) developed a technique to measure the thermal conductivity of an amorphous silicon layer, which was deposited on half of a substrate as shown in Fig 6. Two bismuth bridges were deposited, one each on the layer and the bare substrate. An antimony bridge was deposited over the two bismuth bridges as shown, yielding two thermocouples. The portion of the sample in the dashed rectangle in Fig 6 was coated to enhance radiation absorption. The thermocouple junctions were heated sequentially using a disc of laser light with radius $r_c = 55$ μm. For each case, all of the laser light was incident on the junction. The temperature rise due to the laser light was measured for each case. The ratio of the temperature rise of the thermocouple above the sample layer and the temperature rise of the thermocouple above the bare substrate is U_T. The substrate was modelled as a semi-infinite medium with thermal resistance $R_{sub} = (\pi/4)(r_c/k_{sub})$, corresponding to heat flow in a semi-infinite medium originating from an isothermal disc of radius r_c. The thermal resistance of the sample layer is $d/k_{n,eff}$, and the thermal resistance between the thermocouple junction and the sample surface is d_T/k_T, where d_T and k_T are the thickness and thermal conductivity of the bismuth layer, respectively. The effective layer thermal conductivity $k_{n,eff}$ can be determined by equating the ratio of the temperature changes to the ratio of the summed resistances,

$$U_T = \frac{R_{sub} + \dfrac{d_T}{k_T} + \dfrac{d}{k_n}}{R_{sub} + \dfrac{d_T}{k_T}} \qquad (6)$$

This technique does not require knowledge of the absorbed power. The calculation of R_{sub} requires accurate knowledge of the laser-beam diameter, which resulted in a +/- 9 percent error in the measurement of Goldsmid et al (1983). The uncertainty due to modelling the substrate as a semi-infinite medium and due to lateral conduction in the thermocouple bridges needs to be assessed.

The technique of Cahill et al (1989) was originally developed for measuring the thermal boundary resistance between metal layers and dielectric substrates at low temperatures (Swartz and Pohl, 1987), but can also measure $k_{n,eff}$ of a layer deposited between the metal and the substrate. Figure 7 shows a cross section of the test structure. Two long, parallel metal thermometer bridges were deposited on the sample layer, each of width about $w = 1$ μm, separated by about $x_s = 1$ μm. Bridge A carried a large current density, serving as a heater, while bridge C carried a low current density and experienced negligible Joule heating. The energy dissipated in bridge A traveled through the sample layer, resulting in a heater-substrate temperature difference. The temperature T_A of the heater bridge was determined from its electrical resistance. Bridge C measured T_C, from which T_B was calculated by modelling the substrate as a semi-infinite medium. The total power dissipated in bridge A is Q, and the width and length of bridge A are w and L. The effective thermal conductivity for conduction normal to the layer is

$$k_{n,eff} = \frac{Q \, d}{w \, L \, (T_A - T_B)} \qquad (7)$$

The approximations employed in the thermal analysis are important when the temperature difference in the substrate beneath the two bridges is comparable to the temperature difference normal to the sample layer. This occurs in very thin layers near room temperature, where the substrate thermal conductivity is lower than at low temperatures.

Goodson et al (1993b; 1993a) developed a similar technique for measurements on silicon-dioxide layers above room temperature. The relatively small thermal resistance of these layers at room temperature motivated a more detailed analysis of conduction in the substrate. The contribution of the approximations in the analysis to the experimental uncertainty was estimated by comparing the predicted and measured differences between the temperatures of bridge C and a second non-heating bridge not shown in Fig 7. The separation x_s between bridges A and C was optimized by balancing the competing goals of minimizing the effect of conduction *along* the sample layer and minimizing the temperature difference T_B - T_C. The relative uncertainty in $k_{n,eff}$ was shown to be less than 12 percent for silicon-dioxide layers thicker than 0.3 μm, and less than 20 percent

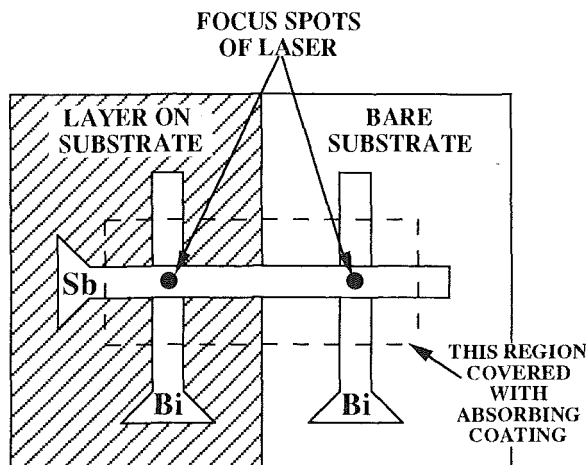**FOCUS SPOTS OF LASER**

LAYER ON SUBSTRATE

BARE SUBSTRATE

Sb

Bi   Bi

THIS REGION COVERED WITH ABSORBING COATING

FIG 6. Top view of the experimental structure of Goldsmid et al (1983) for measuring $k_{n,eff}$.

for layers thicker than 0.03 μm. This experimental accuracy allowed Goodson et al (1993a) to report the annealing-temperature dependence of the thermal conductivity of CVD silicon-dioxide layers of the thicknesses found in highly-integrated electronic circuits.

Schafft et al (1989) and Brotzen et al (1992) also used metal bridges as Joule heaters when they measured $k_{n,eff}$ of silicon-dioxide layers. Both techniques employ only a single bridge. The temperature at the interface between the silicon dioxide layer and the silicon substrate was obtained by an analysis of heat conduction in the entire substrate. For the case of Schafft et al (1989), who measured the conductivities of 1.7 and 3 μm layers using a 3.4 μm wide heater bridge, this resulted in little error because the measured thermal resistance of the silicon dioxide was more than twenty times that of the substrate. As in the technique of Cahill et al (1989), the heater bridge was a resistance thermometer. Since the ratio w/d was of the order of unity, the two-dimensional heat-conduction equation was solved in the sample layer to determine its thermal conductivity. The assumptions used to solve the heat-conduction equation in the substrate would need further investigation if this technique were applied to thinner layers.

Brotzen et al (1992) measured $k_{n,eff}$ of layers of thickness down to 0.1 μm using heater bridge that was 190 μm wide. The temperature difference in their substrate was significant because the sample layer was thinner and the ratio w/d_{sub}, where w is the heater bridge width, was much larger than in the measurements of Schafft et al (1989). Increasing this ratio increases the thermal resistance of the substrate. Brotzen et al (1992) assumed that the aluminum heat sink was isothermal and that the boundary resistance between the heat sink and the substrate was negligible. These assumptions are questionable because of the large value of w/d_{sub}, which was of the order of unity, and may have resulted in an overprediction of the temperature at the bottom interface of the sample layer and an underprediction of $k_{n,eff}$.
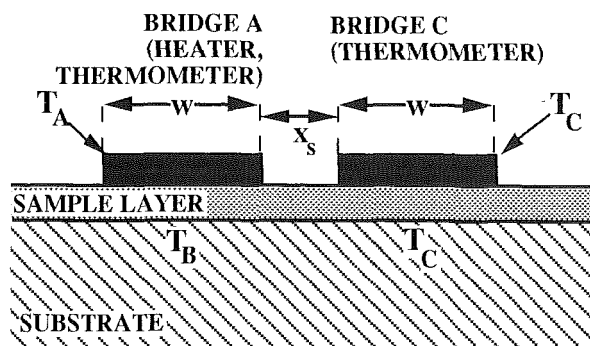
These assumptions are important because Brotzen et al (1992) reported values of $k_{n,eff}$ which are much smaller than those measured in bulk samples. It is not yet possible to determine whether this difference was due to a different layer property or to the approximations in the thermal-conduction analysis. The technique of Cahill et al (1989) is more accurate than those of Schafft et al (1989) and Brotzen et al (1992) because the temperature under the sample layer is calculated using the substrate temperature measured nearby. As a result, $k_{n,eff}$ is less sensitive to approximations in the thermal analysis.

Lambropoulos et al (1989) modified the thermal comparator technique, which was developed by Powell (1957) to measure the thermal conductivity of bulk materials, to measure $k_{n,eff}$ for layers on substrates. A sensing finger and thermocouple apparatus were mounted in a copper heating block as shown in Fig 8. One junction of the thermocouple was at the tip of the finger while the other was inside the block. The temperatures of the copper block and the sample were maintained at 329 and 309 K, respectively. During the measurements, the finger was pressed against the sample with a controlled force and the steady-state thermocouple voltage was recorded. The samples were assumed to be well modelled as semi-infinite media. The temperature of the sensing tip decreased and the thermocouple voltage increased with the increasing thermal conductivities of the sample materials. Measurements performed on bulk samples with known thermal conductivities yielded a calibration curve from which the apparent thermal conductivity of a sample, k_{app}, could be obtained from the thermocouple voltage. For a layer on a substrate, $k_{n,eff}$ can be calculated by assuming the thermal resistance of the substrate is $R_{sub} = (\pi/4)(r_c/k_{sub})$, the relation used by Goldsmid (1983). The difference between R_{sub} and the apparent resistance $R_{app} = (\pi/4)(r_c/k_{app})$ is due to the layer resistance $d/k_{n,eff}$, yielding

$$k_{n,eff} = \frac{4\,d}{\pi\,r_c}\left(\frac{1}{k_{app}} - \frac{1}{k_{sub}}\right)^{-1} \qquad (8)$$

BRIDGE A   BRIDGE C
(HEATER,   (THERMOMETER)
THERMOMETER)

FIG 7. Cross section of the test structure of Cahill et al (1989) for measuring $k_{n,eff}$. Goodson et al. (1993a; 1993b) used a second non-heating bridge to determine the experimental uncertainty for measurements on very-thin layers above room temperature.

This expression is valid when $r_c \gg d$ is satisfied.

The uncertainty in the radius r_c of the contact between the finger and the sample is very important. Powell (1957) showed that the thermocouple voltage was very sensitive to the applied force due to the dependence of the contact area on the force. The contact area also varied with the hardness and elastic properties of the material. While this resulted in less than 6 percent error for many materials, including aluminum, steel, and silicon dioxide, data for lead fell far from the calibration curve established using these materials. The technique of Powell (1957) required r_c to be the same during calibration and measurement, but did not need to determine its magnitude. In contrast, the method of Lambropoulos et al (1989) for layers required both a constant value of r_c and the knowledge of its magnitude, which appears in Eq (8). Thus, the uncertainty due to the differences in the hardnesses of materials is augmented by the uncertainty in the contact radius magnitude. Lambropoulos et al (1989) reported an 80 percent relative uncertainty in r_c,

which was estimated using elastic contact theory. After performing measurements on a variety of dielectric layers, they concluded that in almost all cases the layer thermal conductivity was significantly lower than the bulk conductivity. In many cases, however, the bulk values of the conductivity fell within the experimental uncertainty of the values measured for the layers. This method is attractive because it can be applied without specially-fabricated structures. Accurate measurements require more research to reduce the uncertainty of the contact radius.

Nonnenmacher and Wickramsinghe (1992) measured relative changes of the local thermal conductivity near the surface of a sample using an atomic force microscope (AFM). The AFM maintains a constant force between a probe tip and the surface of a sample and allows atomic-scale profiling of the surface. A heat flow from the tip to the sample was induced by a laser incident on the probe near the tip. The temperature of the probe tip was reduced by thermal conduction to the sample and depended strongly on the local sample conductivity. Variations in the temperature of the probe tip were assumed to be proportional to variations of the potential of the contact between the platinum-coated tip and the tungsten-coated sample surface. The measurement of the contact potential using the AFM was described by Nonnenmacher et al (1991). This technique is attractive because of its unprecedented spatial resolution, which is governed by the radius of the contact between the probe tip and the layer surface. But quantitative measurements of the thermal conductivity will require precise knowledge of the contact radius, the heat flux, and the derivative with respect to temperature of the contact potential. The heat-transfer induced by this high-resolution method closely resembles that in the thermal-comparator technique of Powell (1957).

3.2   Transient Techniques

There are two types of transient techniques which measure the thermal conductivity normal to layers. Techniques of the first type use periodic heating on the surface of the layer, and determine the thermal conductivity from the periodic temperature at the layer surface. The most common version of this approach is the 3-ω technique of Cahill et al (1989), who applied to thin layers the technique developed for bulk amorphous solids by Cahill and Pohl (1987) and Cahill (1990). A thin, narrow metal bridge was deposited on the layer-substrate composite. The bridge served both as a heater and as an electrical-resistance thermometer. The bridge carried a sinusoidal current of angular frequency ω. The rate of heat generation in the bridge was proportional to the square of the current and had the frequency 2ω. The inverse thermal diffusion length for this case is $\lambda_T = (2\omega/\alpha)^{1/2}$. The amplitude of the temperature oscillations in the layer is approximately (Carslaw and Jaeger, 1959)

$$T_a(r) = \frac{q_{al}}{\pi\,k}\,K_0\left(\lambda_T r\right) \qquad (9)$$

where K_0 is the modified Bessel function of order zero, r is the distance from the heating source, and q_{al} is the amplitude of the energy deposited in the bridge per unit time and length. This assumes that the bridge is a line source and neglects variation of the properties of the semi-infinite medium with depth. The frequency of the temperature oscillations is equal to that of the driving source, 2ω. For $\lambda_T\,r \ll 1$, the temperature amplitude approximately satisfies

$$\frac{\partial T_a(r)}{\partial\left[\ln(\omega)\right]} = -\frac{q_{al}}{2\,\pi\,k} \qquad (10)$$

The electrical resistance of the bridge varies linearly with temperature. The voltage along the bridge is the product of the applied bias current, periodic with frequency ω, and the bridge electrical resistance, periodic with frequency 2ω. This yields a component of the voltage signal with frequency 3ω whose amplitude is related to the amplitude of the periodic bridge temperature. The thermal conductivity can be obtained using Eq (10) and the bridge-temperature amplitude at two frequencies.

This technique has the advantage of allowing the thermal conductivity to be probed within a targeted thickness of the layer-substrate composite. For $d\,\lambda_T \ll 1$, where d is the layer thickness, it measures the properties of the substrate. For $d\,\lambda_T \gg 1$, it yields the properties of the layer. The depth resolution of the technique is limited by the requirement of cylindrical symmetry about the heater bridge, $\lambda_T\,w \ll 1$, where w is the width of the bridge. The available fabrication technology makes possible measurements on layers of minimum thickness 10 μm. For the opposing limit, $\lambda_T\,w \gg 1$, the conduction is approximately one-dimensional in the direction normal to the layer. The possibility of using this limit to measure the thermal conductivity of thinner layers should be explored. This approach would allow the technique to sensitively measure the thermal boundary resistance between the layer and the heater bridge.
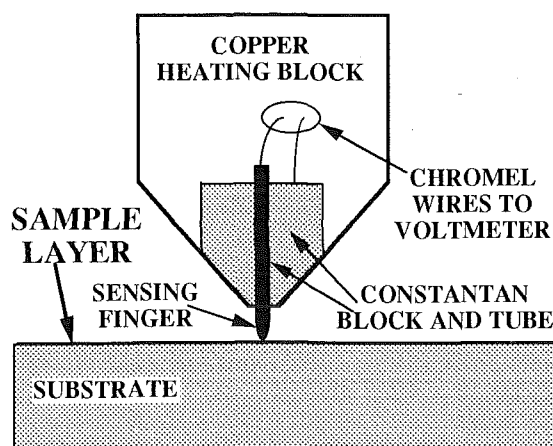
FIG 8.   Schematic of the thermal comparator used by Lambropoulos et al (1989).

The value of the thermal conductivity obtained using the 3-ω technique is not necessarily equal to $k_{n,eff}$. If the thermal conductivity is anisotropic, the radial symmetry of the technique yields a conductivity which is a function of the thermal conductivities both normal to and along the layer. The measured conductivity is most affected by regions near the surface of the layer.   As a result, the thermal conductivity obtained using the 3-ω method may not be appropriate for analyzing steady-state conduction normal to thin-layered structures. The influence of thermal boundary resistances on this technique has not been assessed. These could be important, particularly at low temperatures.

The second type of transient technique for measuring the thermal conductivity normal to layers is non-contact and applies to free-standing layers. A brief pulse of laser energy is applied at the layer surface, and the thermal conductivity is calculated from the transient temperature of the opposite layer surface, measured using IR thermography.   This approach was recently applied to amorphous polymer layers by Tsutsumi and Kiyotsukuri (1988), to metals by Shibata et al (1991), and to diamond layers by Graebner et al (1992b). Like the optical techniques discussed in Section 2.2, this one benefits from the deposition of optically-absorbing layers on the front and rear surfaces. The thermal resistances and heat capacities per unit area of these layers can be made small compared to those of the sample layer. For a sheet of laser light incident on one surface of the layer, the analysis of one-dimensional conduction through the layer yields the approximate temperature rise at the opposite side at time t after the energy deposition (Graebner et al, 1992b),

$$\Delta T(t) = \frac{E''}{Cd}\left[1 + 2\sum_{i=1}^{\infty}(-1)^n \exp\left(\frac{-i^2\pi^2}{d^2}\alpha\ t\right)\right] \quad (11)$$

where α is the thermal diffusivity in the direction normal to the layer, E'' is the energy deposited per unit area and C is the specific heat per unit volume.   Fitting the measured response of the detector to Eq (11) yields α. Both the period of the pump laser and the response time of the detector need to be much smaller than d^2/α. This limits the practical application of this technique relatively thick layers.   For 5 μm amorphous layers at room temperature, d^2/α is of the order of 25 μs, which requires a rather fast IR detector. For highly-conductive layers, such as silicon and diamond, the application of this technique is limited to layers several tens of micrometers thick. The possibility of using an electrical-resistance thermometer bridge, deposited on the back surface of the layer, should be investigated. This would decrease the response time and allow thinner layers to be measured.

4.   SUMMARY AND RECOMMENDATIONS

The techniques measuring the thermal conductivity along layers are summarized in Table I.   The periodic optical techniques of Harata et al (1990), Käding et al (1993a; 1993b), Hatta (1985), and Visser et al (1992) provide attractive alternatives to the standard steady-state technique of Pompe and Schmidt (1973). The application of these optical methods to very thin layers requires additional research on the impact of absorbing coatings. Tai et al (1988), and Mastrangelo and Muller (1988) and Völklein and Baltes (1992) developed novel microstructures for thermal conductivity measurements that closely resembled the structure whose design required the property.

Techniques measuring the effective thermal conductivity normal to layers are summarized in Table II. Goldsmid (1983) developed a useful technique whose uncertainty needs further investigation. The technique of Cahill et al (1989) is the most accurate because it comes the closest to measuring the temperature on both sides of the sample layer. The technique of Lambropoulos et al (1989), which employs a thermal comparator, is an easily-applied nondestructive technique, but requires more work to precisely determine the contact area. The 3-ω technique of Cahill et al (1989) allows a targeted depth within the layer to be probed, but is limited to layers thicker than about 10 μm.

More detailed uncertainty analysis is required for most of the techniques available. Uncertainty analysis is made more important by the large heat fluxes present in thin layers in microelectronic circuits.   A relatively small error in the measured conductivity of a layer can cause a large absolute temperature error, resulting in inaccurate predictions of the circuit performance and reliability.

The microstructures of thin layers depend strongly on the fabrication techniques used to make them.   Thermal conductivity measurements must therefore be performed using microstructures fabricated using the same processes as those of the real devices for which the thermal conductivity is needed.

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Table I. Summary of techniques measuring the effective thermal conductivity along layers.

Geometry	Tested Temp. Range	Heating Time-Dependence and Source	Temperature Measurement	References
free-standing bridge	10-450 K	steady-state heater	thermocouple	Nath and Chopra (1973); Morelli et al (1988)
free-standing electrically conducting bridge	80-700 K	steady-state or transient Joule heating	sample-bridge electrical resistance	Völklein and Kessler (1984); Mastrangelo and Muller (1988); Boiko et al (1973); Tai et al (1988)
substrate etched from beneath portion of layer	near 300 K	steady-state heater-bridge	microfabricated thermocouples	Graebner et al (1992c)
free-standing bridge	near 300 K	periodic laser sheet incident on half of bridge	thermocouple	Hatta (1985)
free-standing bridge	near 300 K	laser pulse, line focus	IR thermography	Shibata et al (1991)
free-standing layer	near 300 K	periodic laser beam with point focus	IR thermography	Visser et al (1992)
layer with or without substrate	near 300 K	two pulsed laser beams that interfere on surface	deflection of probe laser beam	Harata et al (1990); Käding et al (1993a)

Table II. Summary of techniques measuring the effective thermal conductivity normal to layers.

Layer Geometry	Tested Temp. Range	Heating Time-Dependence and Source	Temperature Measurement	References
dielectric layer on substrate	near 300 K	steady-state laser with disc-shaped focus	thermocouple bridges	Goldsmid et al (1983)
dielectric layer on substrate	10-400 K	steady-state heating in microfabricated bridge	bridge resistance thermometers	Cahill et al (1989); Schafft et al (1989); Goodson et al (1993a; 1993b)
dielectric layer on substrate	near 300 K	steady-state heating in microfabricated bridge	thermocouple on heater	Brotzen et al (1992)
layer on substrate	near 300 K	steady-state heating in copper block	thermocouple	Lambropoulos et al (1989)
layer with or without substrate	near 300 K	steady-state heating through AFM probe tip	contact potential at AFM probe tip	Nonnenmacher and Wickramsinghe (1992)
dielectric layer with or without substrate	1 - 300 K	periodic heating in a microfabricated bridge	bridge resistance thermometer	Cahill et al (1989)
free-standing layer	near 300 K	pulsed sheet of laser light	IR thermography	Graebner et al (1992b); Shibata et al (1991); Tsutsumi and Kiyotsukuri (1988)

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